SYNTHESIS AND PROPERTIES OF SUBSTITUTED 1-(2-BENZOTHIAZOLYL)-2-PYRIDONES

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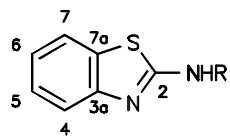
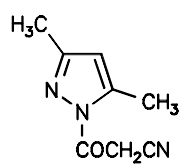
The studied 1-(2-benzothiazolyl)-2-pyridones *Va–Vf* were prepared from *N*-(2-benzothiazolyl)-cyanoacetamide (*II*) which on reaction with 4-substituted benzaldehydes afforded 3-aryl-*N*-(2-benzothiazolyl)-2-cyano-2-propenamides *IVa–IVg*. Compounds *IVa–IVf* were cyclized with malonodinitrile in the presence of piperidine to give the corresponding pyridones *Va–Vf*.

The significant biological properties and utilization of various benzothiazoles[1,2] on the one hand and pyridones[3,4] on the other have influenced the research in these two regions. In this context, derivatives containing both the mentioned moieties in the molecule are also of interest.

The present communication concerns the synthesis and study of properties of some substituted 1-(2-benzothiazolyl)-2-pyridones *Va–Vf* which were synthesized starting from 2-aminobenzothiazole[5] (*I*).

1-Cyanoacetyl-3,5-dimethylpyrazole[6] (*III*), an effective reagent for cyanoacetylation of various amino derivatives[7], reacted with amine *I* in boiling toluene to give the corresponding cyanoacetamide *II* in 91% yield.





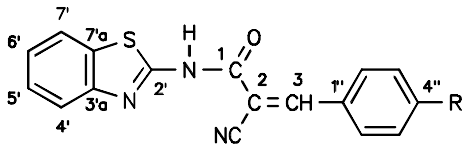
III

	R
I	H
II	$COCH_2CN$

3-Aryl-*N*-(2-benzothiazolyl)-2-cyano-2-propenamides *IVa–IVg* were obtained in very good yields (75–95%; see Table I) by Knoevenagel reaction of the *C*-acid *II* with 4-substituted benzaldehydes. The condensation was performed at elevated temperature in 10% ethanolic sodium hydroxide or in boiling solution of potassium acetate in acetic acid.

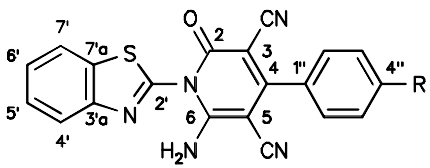
As shown below, 2-propenamides of general formula *IV* are suitable precursors for the synthesis of polysubstituted 2-pyridones *V*. Although many authors prepared 2-pyridones in various ways[8–10], a synthesis starting from derivatives *IV* has not been described so far.

The desired 6-amino-4-aryl-1-(2-benzothiazolyl)-3,5-dicyano-2-pyridones *Va–Vf* were prepared by treatment of *N*-(2-benzothiazolyl)-2-propenamides *IVa–IVf* with malonodinitrile in boiling ethanol in the presence of piperidine (yields 17–34%). The reac-



IV

	R		R
a	H	*e*	NO_2
b	CH_3	*f*	CN
c	OCH_3	*g*	$N(CH_3)_2$
d	Cl		



V

	R		R
a	H	*d*	Cl
b	CH_3	*e*	NO_2
c	OCH_3	*f*	CN

TABLE I
Melting points, yields and analytical data for compounds *IVa–IVg* and *Va–Vf*

Compound	M.p., °C Yield, %	Formula (M.w.)	Calculated/Found			
			% C	% H	% N	% S
IVa	221–223[a]	$C_{17}H_{11}N_3OS$	66.87	3.63	13.76	10.50
	95	(305.3)	67.67	3.47	13.98	10.71
IVb	235–236[a]	$C_{18}H_{13}N_3OS$	67.69	4.10	13.16	10.04
	75	(319.4)	66.96	4.24	13.18	10.20
IVc	236–237[a]	$C_{18}H_{13}N_3O_2S$	64.46	3.91	12.53	9.56
	90	(335.4)	63.70	3.96	12.50	9.22
IVd	239–241[b]	$C_{17}H_{10}ClN_3OS$	60.09	2.97	12.37	9.44
	77	(339.8)	59.52	3.02	12.47	9.34
IVe	280–282[b]	$C_{17}H_{10}N_4O_3S$	58.28	2.88	15.99	9.15
	91	(350.3)	58.53	2.85	16.23	9.39
IVf	280–282[a]	$C_{18}H_{10}N_4OS$	65.44	3.05	16.96	9.71
	91	(330.4)	64.67	3.12	17.00	9.99
IVg	292–294[c]	$C_{19}H_{16}N_4OS$	65.49	4.63	16.08	9.20
	86	(348.4)	64.85	4.72	16.31	9.33
Va	299–302[b]	$C_{20}H_{11}N_5OS$	65.03	3.00	18.96	8.68
	34	(369.4)	65.46	3.12	19.05	8.63
Vb	299–301[b]	$C_{21}H_{13}N_5OS$	65.78	3.42	18.27	8.36
	17	(383.4)	65.52	3.41	18.42	8.47
Vc	308–311[b]	$C_{21}H_{13}N_5O_2S$	64.15	3.28	17.53	8.03
	17	(399.4)	63.59	3.28	17.80	8.13
Vd	315–317[c]	$C_{20}H_{10}ClN_5OS$	59.48	2.50	17.34	7.94
	25	(403.8)	59.49	2.76	17.57	7.60
Ve	306–309[c]	$C_{20}H_{10}N_6O_3S$	57.96	2.43	20.28	7.74
	32	(414.4)	57.79	2.54	20.38	7.79
Vf	306–311[c]	$C_{21}H_{10}N_6OS$	63.92	2.56	21.31	8.13
	33	(394.4)	64.28	2.68	21.52	8.00

Crystallized from: [a] ethanol, [b] acetic acid, [c] *N*,*N*-dimethylformamide.

TABLE II

Infrared (in KBr) and UV spectra (in dioxane, c 1 . 10^{-4} mol l^{-1}) of compounds *II*, *IVa–IVg* and *Va–Vf*

Compound	IR spectrum, cm^{-1}			UV spectrum	
	ν(NH)[a]	ν(C≡N)	ν(C=O)	λ_{max}, nm	log ε
II	3 293	2 261	1 690	220	3.30
				275	3.14
				298	2.94
IVa	3 177	2 226	1 669	222	3.40
				334	3.30
IVb	3 320	2 218	1 688	222	3.39
				340	3.44
IVc	3 248	2 220	1 676	218	3.42
				360	3.60
IVd	3 290	2 218	1 684	223	3.40
				335	3.40
IVe	3 175	2 238	1 690[b]	217	3.31
				321	3.13
				363	3.14
IVf	3 351	2 231	1 688[b]	217	3.46
		2 228		306	3.40
				358	3.34
IVg	3 274	2 216	1 673	218	3.40
				263	3.09
				321	2.80
				434	3.67
Va	3 319	2 210	1 676	216	3.58
				276	3.51
				305[c]	3.11
				381	3.08
Vb	3 318	2 209	1 676	217	3.59
				279	3.48
				308[c]	3.28
				381	3.05
Vc	3 312	2 215	1 676	217	3.61
				278	3.44
				331	3.35
				375[c]	3.06
Vd	3 326	2 224	1 678	217	3.58
		2 210		278	3.50
				310[c]	3.14
				379	3.05

TABLE II
(*Continued*)

Compound	IR spectrum, cm^{-1}			UV spectrum	
	ν(NH)[a]	ν(C≡N)	ν(C=O)	λ_{max}, nm	log ε
Ve	3 304	2 218	1 680	217	3.62
				273	3.60
				305[c]	3.25
				382	3.09
Vf	3 301	2 218	1 678	217	3.57
				268	3.49
				310[c]	3.03
				381	3.03

[a] For compounds *Va–Vf* $\nu(NH_2)$; [b] significantly lower band intensity; [c] shoulder.

TABLE III
^{1}H NMR data (δ, ppm; *J*, Hz) of compounds *IVa–IVg* in $(CD_3)_2SO$

Compound	NH	H-3	H-4′ *J*(4′,5′)	H-7′	H-5′ *J*(5′,6′)	H-6′ *J*(6′,7′)	Other signals
IVa	9.96 s	8.49 s	7.96 d 7.9	[a]	7.48 t 7.8	7.34 t 7.3	8.06–7.98 m and 7.70–7.55 m, 2 H and 4 H (phenyl and H-7′ of 2-benzothiazolyl)
IVb	9.90 s	8.37 s	7.88 d 8.3	7.61	7.46 t 8.3	7.32 t 8.3	7.88 d and 7.36 d, 2 H and 2 H, *J* = 8.3 (4-methylphenyl); 2.36 s, 3 H (CH_3)
IVc	9.81 s	8.40 s	7.90 d 7.9	7.63 d	7.45 t 7.4	7.31 t 7.9	8.03 d and 7.12 d, 2 H and 2 H, *J* = 8.9 (4-methoxyphenyl); 3.08 s, 3 H (OCH_3)
IVd	9.95 s	8.47 s	7.98 d 8.1	7.62 d	7.50 t 7.2	7.36 t 7.7	8.05 d and 7.69 d, 2 H and 2 H, *J* = 8.6 (4-chlorophenyl)
IVe	–	8.57 s	7.96 d 7.9	7.64 d	7.51 t 7.3	7.37 t 7.7	8.39 d and 8.22 d, 2 H and 2 H, *J* = 8.9 (4-nitrophenyl)
IVf	–	8.52 s	7.98 d 7.7	7.61 d	7.50 t 7.1	7.35 t 7.7	8.16 d and 8.06 d, 2 H and 2 H, *J* = 8.2 (4-cyanophenyl)
IVg	9.63 s	8.24 s	7.89 d 7.9	7.64 d	7.44 t 7.0	7.30 t 7.7	7.91 and 6.83 d, 2 H and 2 H, *J* = 9.1 (4-dimethylaminophenyl); 3.06 s, 3 H ($(CH_3)_2N$)

[a] Multiplet 7.70–7.55, 4 H (H-3″, H-4″, H-5″ of phenyl and H-7′ of 2-benzothiazolyl).

tion proceeds probably as the Michael addition of malonodinitrile to the α,β-unsaturated system followed by cyclization.

The structure of *N*-(2-benzothiazolyl)cyanoacetamide (*II*), substituted 2-propenamides *IVa–IVg* and 1-(2-benzothiazolyl)-2-pyridones *Va–Vf* was confirmed by their spectral data and elemental analyses (Tables I–VI). In addition to the characteristic bands, the infrared spectra of compounds *IVa–IVg* show also a weak band at 1 591–1 614 cm^{-1} due to C=C bond (Table II). The amide hydrogen in derivatives *IVe* and *IVf* is apparently more acidic and the compounds are partly enolized; this results in the observed lower intensity of the ν(C=O) band.

The extended conjugated system in compounds *IVa–IVg* (as compared with *N*-(2-benzothiazolyl)cyanoacetamide (*II*)) manifests itself in the UV spectra (Table II) not only by a bathochromic shift of the maxima (36–136 nm, according to the substituent) but also by increased intensity of the bands. The pyridone grouping in derivatives *Va–Vf* is characterized by an absorption maximum in the region 375–382 nm (shifted to the visible region only by 15–49 nm relative to the corresponding derivatives *IVa–IVf*).

The ^{1}H NMR data confirm unequivocally the structure of the synthesized compounds. In the spectra of substituted 2-propenamides *IVa–IVg* (Table III) the olefinic

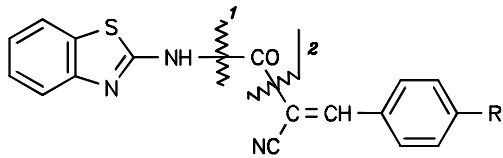
TABLE IV
^{1}H NMR data (δ, ppm; *J*, Hz) of compounds *Va–Vf* in $(CD_3)_2SO$

Compound	NH_2	H-4′ *J*(4′,5′)	H-7′ *J*(6′,7′)	Other signals
Va	8.71 bs	8.22 d 7.4	8.12 d 7.8	7.70–7.55 m, 7 H (H-5′ and H-6′ of 2-benzothiazolyl and phenyl)
Vb	8.70 bs	8.22 d 8.0	8.12 d 7.3	7.68–7.55 m, 2 H (H-5′ and H-6′ of 2-benzothiazolyl); 7.48 d and 7.40 d, 2 H and 2 H, *J* = 7.9 (4-methylphenyl); 2.43 s, 3 H (CH_3)
Vc	8.69 bs	8.22 d 8.2	8.12 d 7.7	7.67–7.58 m, 2 H (H-5′ and H-6′ of 2-benzothiazolyl); 7.56 d and 7.15 d, 2 H and 2 H, *J* = 8.7 (4-methylphenyl); 3.88 s, 3 H (OCH_3)
Vd	8.79 bs	8.22 d	8.12 d 7.6	7.73 d and 7.65 d, 2 H and 2 H, *J* = 8.7 (4-chlorophenyl); 7.65–7.56 m, 2 H (H-5′ and H-6′ of 2-benzothiazolyl)
Ve	8.82 bs	8.23 d 7.1	8.12 d 7.2	8.43 d and 7.88 d, 2 H and 2 H, *J* = 8.7 (4-nitrophenyl); 7.68–7.56 m, 2 H (H-5′ and H-6′ of 2-benzothiazolyl)
Vf	8.82 bs	8.22 d 7.1	8.13 d 7.5	8.07 d and 7.79 d, 2 H and 2 H, *J* = 7.7 (4-cyanophenyl); 7.72–7.52 m, 2 H (H-5′ and H-6′ of 2-benzothiazolyl)

proton H-3 appears as a singlet at δ 8.57–8.24. Proton spectra of 1-(2-benzothiazolyl)-2-pyridones *Va–Vf* (Table IV) exhibit a broad signal at δ 8.82–8.69 due to protons of the amino group. As expected, the highest effect of the substituent R was on the signals of phenyl protons H-2″, H-6″ and H-3″, H-5″ at δ 7.88–7.48 and 8.64–7.15, respectively. Practically no change was observed for the benzothiazole protons H-4′ and H-7′ (δ 8.23–8.22 and 8.13–8.12, respectively). Analogously, in the ^{13}C NMR spectra of compounds *Va–Vf* (Table V) the highest effect of substituent R was observed on carbon atoms of the ring to which the substituent was attached.

The principal fragmentation pattern in the mass spectra of compounds *IVa–IVg* (Table VI) are depicted in Scheme 1.

The molecular ion peaks in the mass spectra of pyridones *Va–Vf* (Table VI) belong to the most intensive ones. For most of these derivatives (except *Vc* and *Ve*), ions $[M - \overset{\bullet}{H}]^+$ represent the base peaks. On the other hand, the loss of water from the molecular ions contributes only very little (less than 10%) to the fragmentation pattern. The relative abundance of fragment ions formed by loss of carbon monoxide from $M^{+\bullet}$ ions amounts to 10–20%. Within this range of relative intensities are present also the benzothiazole ions *m/z* 135, arising by fission of the C–N bond and hydrogen transfer. Mass spectra of all the pyridone derivatives exhibit also fragment ions of *m/z* 177 and *m/z* 150, formed by fission of bonds in the pyridine ring. The loss of the substituent R from the molecular ion is marked only in the case of the nitro group (*Vf*) where it gives rise to the ion of *m/z* 369 (49%).



	R	$M^{+\bullet}$, *m/z* (%)	*1*, *m/z* (%)	*2*, *m/z* (%)
IVa	H	305 (76)	156 (100)	128 (87)
IVb	CH_3	319 (76)	170 (100)	142 (26)
IVc	OCH_3	335 (63)	186 (100)	158 (37)
IVd	Cl	339 (73)	190 (100)	162 (53)
IVe	NO_2	350 (100)	201 (29)	–
IVf	CN	330 (95)	181 (69)	153 (100)
IVg	$N(CH_3)_2$	348 (25)	199 (100)	171 (35)

SCHEME 1

EXPERIMENTAL

The melting points were determined on a Kofler block. IR spectra were recorded on a FTIR PU9800 (Philips) spectrometer, UV spectra (λ in nm, ε in $m^2\ mol^{-1}$) on a Specord UV-VIS M-40 (Zeiss, Jena) instrument. Proton and ^{13}C NMR spectra were measured on a Varian VXR-300 spectrometer (300 MHz for ^1H and 60 MHz for ^{13}C) in hexadeuteriodimethyl sulfoxide with tetramethylsilane as internal standard. Chemical shifts are given in ppm (δ-scale), coupling constants *J* in Hz). The ^{13}C NMR spectra were assigned using the SINEPT and HETCOR techniques and a set of CHEM3WIND programs. Mass spectra (EI) were taken on an MS 902 S (A.E.I. Manchester) spectrometer; direct inlet, electron energy 70 eV, trap current 100 μA, ion source temperature 220–240 °C (for *Va–Vf*) or 150–200 °C (for *II*, *IVa–IVg*).

N-(2-Benzothiazolyl)cyanoacetamide (*II*)

A solution of 1-cyanoacetyl-3,5-dimethylpyrazole[6] (*III*; 16.3 g, 100 mmol) in anhydrous toluene (150 ml) was added to a solution of 2-aminobenzothiazole[5] (*I*; 15.0 g, 100 mmol) in toluene (200 ml) and the mixture was refluxed for 4 h. After cooling, the deposited solid was collected and crystallized from acetic acid, yield 19.7 g (91%), m.p. 246–248 °C. For IR, UV and mass spectra see Tables II and VI. ^1H NMR spectrum: 7.93 d, 1 H (H-4, *J*(4,5) = 7.9, *J*(4,6) = 1.3); 7.73 d, 1 H (H-7, *J*(7,6) = 7.9,

TABLE V
^{13}C NMR data (δ, ppm; *J*, Hz) of compounds *Va–Vc*, *Ve–Vf* in $(CD_3)_2SO$

Compound[a]	^{13}C NMR spectum								
	C-2	C-3	C_3-CN	C-4	C-5	C_5-CN	C-6	C-2	C-3′a
Va	156.5	75.8	114.6	162.1	88.0	115.0	154.1	158.7	149.2
Vb	156.5	75.8	114.8	162.1	87.6	115.2	154.1	158.8	149.2
Vc	156.5	75.8	114.9	160.8	87.4	115.3	154.2	158.8	149.2
Ve	156.6	75.6	114.3	160.1	87.6	114.7	153.8	158.5	149.4
Vf	156.6	75.6	114.3	160.4	87.6	114.8	153.9	158.5	149.4
	C-4′	C-5′	C-6′	C-7′	C-7′a	C-1″	C-2″ C-6″	C-3″ C-5″	C-4″
Va	122.2	126.0	126.1	123.3	136.5	134.2	127.4[b]	128.2[b]	130.0
Vb	122.2	126.0	126.1	123.3	136.5	131.3	128.8[b]	127.5[b]	140.0
Vc	122.2	126.0	126.1	123.3	136.4	126.1	129.4	113.8	161.7
Ve	122.3	126.0	126.1	123.3	136.6	140.5	129.2	123.5	148.5
Vf	122.2	126.0	126.2	123.7	136.6	138.8	128.6	123.3	113.0

[a] Additional signals: for *Vb* 20.3 (CH_3), for *Vc* 55.1 (OCH_3), for *Vf* 117.6 (C4″-CN); [b] the signals may be interchanged.

J(7,5) = 1.3); 7.44 dt, 1 H (H-5, *J*(5,6) = 7.6); 7.31 dt, 1 H (H-6); 4.02 s, 2 H (CH_2). ^{13}C NMR spectrum: 163.1 (C=O); 26.38 (CH_2); 115.1 (CN); 157.9 (C-2); 148.2 (C-3a); 120.8 (C-4); 124.2 (C-5); 126.6 (C-6); 121.9 (C-7); 131.6 (C-7a). For $C_{10}H_7N_2OS$ (217.2) calculated: 55.28% C, 3.25% H, 19.34% N, 14.76% S; found: 55.44% C, 3.24% H, 19.40% N, 14.32% S.

General Procedure for Preparation of 3-Aryl-*N*-(2-benzothiazolyl)-2-cyano-2-propenamides *IVa–IVc* and *IVg*

A stirred mixture of 4-substituted benzaldehyde (5 mmol), *N*-(2-benzothiazolyl)cyanoacetamide (*II*; 1.08 g, 5 mmol), anhydrous potassium acetate (1.96 g, 20 mmol) and acetic acid (20 ml) was refluxed for 3 h. After pouring on ice, the crude product was filtered, washed with water, dried and crystallized from an appropriate solvent. For yields, melting points and elemental analyses see Table I.

TABLE VI
Mass spectra (EI) of derivatives *II*, *IVa–IVg* and *Va–Vf*. For each compound 10 most abundant peaks are given.

Compound	*m*/*z* (relat. abundance, %)
II	217($M^{+\bullet}$, 37), 178(8), 151(7), 150(100), 149(9), 123(10), 115(8), 108(7), 69(6), 45(6)
IVa	305($M^{+\bullet}$, 76), 304(21), 277(20), 276(26), 200(24), 156(100), 150(64), 128(87), 101(33), 77(42)
IVb	320(19), 319($M^{+\bullet}$, 76), 318(17), 291(14), 290(15), 170(100), 150(35), 142(26), 116(16), 115(64)
IVc	336(15), 335($M^{+\bullet}$, 63), 334(21), 187(19), 186(100), 158(37), 150(15), 143(12), 115(11), 77(10)
IVd	341(29), 339($M^{+\bullet}$, 73), 338(21), 310(25), 192(33), 190(100), 162(53),150(47), 127(49), 126(37)
IVe	350($M^{+\bullet}$, 100), 322(34), 321(34), 228(43), 201(29), 200(62), 177(35), 155(86), 150(52), 127(44)
IVf	330($M^{+\bullet}$, 95), 329(35), 302(31), 301(44), 228(30), 200(47), 181(69), 153(100), 150(81), 126(40)
IVg	349(6), 348($M^{+\bullet}$, 25), 347(12), 200(16), 199(100), 172(5), 171(35), 156(7), 155(5), 134(8)
Va	370(25), 369($M^{+\bullet}$, 98), 368(100), 341(14), 303(11), 177(12), 165(11), 150(24), 135(17), 96(12)
Vb	384(27), 383($M^{+\bullet}$, 98), 382(100), 355(16), 177(18), 150(34), 149(10), 135(15), 108(11), 96(11)
Vc	400(26), 399($M^{+\bullet}$, 100), 398(90), 371(11), 355(9), 333(8), 177(14), 150(37), 149(9), 135(11)
Vd	405(36), 404(59), 403($M^{+\bullet}$, 93), 402(100), 375(13), 177(17), 150(33), 135(20), 123(14), 96(14)
Ve	415(27), 414($M^{+\bullet}$, 100), 413(81), 383(19), 367(49), 177(27), 176(32), 150(42), 149(17), 135(19)
Vf	395(23), 394($M^{+\bullet}$, 80), 393(100), 366(14), 177(15), 150(29), 149(19), 135(24), 108(15), 96(18)

General Procedure for Preparation of 3-Aryl-*N*-(2-benzothiazolyl)-2-cyano-2-propenamides *IVd–IVf*

A solution of 4-substituted benzaldehyde (5 mmol) in ethanol (20 ml) and 10% aqueous sodium hydroxide (5 drops) were added to a stirred boiling solution of *N*-(2-benzothiazolyl)cyanoacetamide (*II*; 1.08 g, 5 mmol) in ethanol (50 ml). The reaction mixture was kept at reflux for 2 h (compound *IVd*) or 15 min (compounds *IVe* and *IVf*) and then gradually cooled to ambient temperature (5 h) with stirring. The crude product was purified by crystallization from an appropriate solvent. For yields, melting points and elemental analyses see Table I.

General Procedure for Preparation of 6-Amino-4-aryl-1-(2-benzothiazolyl)-3,5-dicyano-2-pyridones *Va–Vf*

A mixture of 3-aryl-*N*-(2-benzothiazolyl)-2-cyano-2-propenamide *IVa–IVf* (3 mmol), malonodinitrile (0.26 g, 4 mmol), piperidine (4 drops) and anhydrous ethanol (60 ml) was refluxed for 6 h. After cooling, the solid was filtered and crystallized from an appropriate solvent. For yields, melting points and elemental analyses see Table I.

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